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January 27, 2014

Ms. Dania Zinner
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TASK ORDER NUMBER 3019
QA SUPPORT FOR THE LIBBY ASBESTOS SITE

Dear Ms. Zinner:

Enclosed please find the Technical Memorandum for the EMSL-Libby TEM Inter-laboratory Study. This memorandum is a deliverable under Task 2 of Task Order 3019.

If you have any questions, please feel free to contact me.

Sincerely,

Jenny Vonnahme for MPL

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Memorandum

From: Michael P. Lenkauskas
Date: January 27, 2014
Subject: EMSL Analytical (Libby, MT) Inter-labs

This memorandum summarizes the results of two separate TEM inter-laboratory studies, one performed between EMSL Analytical in Libby, Montana (EMSL27) and Reservoirs Environmental in Denver, Colorado (RESI), and the other between EMSL27 and Hygeia Environmental in Sierra Madre, California (Hygeia). Both of these studies were conducted to determine the ability of EMSL27 to analyze investigative samples collected from the Libby Superfund Site due to issues that were identified from this laboratory in 2013 by EPA and its contractors. These issues, summarized in a technical memo that the QATS Program prepared and submitted to EPA on September 20, 2013, included:

- TEM Inter-lab sample preparation issues
- Inadequate frequency of project-specific QC analyses
- Possible misidentification of samples
- Result discrepancies between TEM rapid TAT and full analysis of OU3 water samples

Not discussed in the memo was the tendency of analysts at EMSL27 to report the presence of Sodium (Na) and Potassium (K) and assign WRTA (Winchite, Richterite, Tremolite & Actinolite) designations for the majority of structures identified as Libby Amphibole (LA).

On September 25-26, 2013 ESAT Region 8 personnel provided additional Transmission Electron Microscopy (TEM) analysis training to EMSL27 staff. This occurred prior to the TEM Inter-laboratory studies described below. The laboratory is currently ineligible to receive investigative samples from Libby Superfund Site Operable Units (OUs) pending the outcome of these studies.

EMSL27/RESI Inter-laboratory Analysis

On October 23, 2013 ESAT Region 8 directed EMSL27 to prepare one sample (FB-00014) and provide the TEM grid preparation to RESI for inter-laboratory analysis. The sample was to be prepared, analyzed, and assessed based on the most current version of laboratory modification LB-000029. EMSL27 performed the re-preparation and shipped the sample to RESI on 10/31/2013; however, they did not submit the associated paperwork (i.e. structures maps and Grid Opening [GO] selection) to QATS for review, as required in LB-000029. Upon review of the paperwork, which QATS did receive prior to RESI beginning the inter-laboratory analysis, QATS discovered that EMSL27 had not selected the correct GOs to be analyzed. The laboratory selected GOs with non-asbestos materials (NAMS) and Chrysotile (CH) structures instead of those containing the highest number of LA structures as required by LB-000029. EMSL27 was contacted regarding the above discrepancies and was directed to review the re-preparation results, select the appropriate GOs, and provide RESI with the necessary revised documentation, which would allow them to proceed with the inter-laboratory analysis. The corrected paperwork was provided by EMSL27 to RESI on 11/11/2013. The completed inter-lab report and associated EDDs were uploaded to the FTP site by RESI on 1/10/2014. Note that the report delivered by

RESI was outside of the 10 day Turn-Around-Time (TAT) and nine (9) weeks after the November 6, 2013 sample receipt date.

The re-preparation and inter-laboratory results were reviewed by RESI and QATS on a GO by GO basis and a structure by structure basis to determine concordance with the criteria described in LB-000029 (refer to **Table 1**).

Table 1 - Recount Analysis Concordance Rules – LB-000029	
Measurement Parameter	**Concordance Rule**
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10 percent (%) as calculated as RPD (((maximum count – minimum count)/average count)*100%).
Asbestos class of structure (LA, OA, CH)	Must agree 100% on CH vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 1 micron (µm) or 10% (whichever is less stringent). For clusters and matrices, must agree within 2 µm or 20% (whichever is less stringent).
LA Structure width	For fibers and bundles, must agree within 0.5 µm or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.
Presence of Sodium (Na) and Potassium (K)	There is no rule for concordance, but must be tabulated to identify potential trends that may indicate inconsistencies in recording practices or interpretation of spectra.

A summary of the FB-00014 sample inter-lab results are provided below by assessment comparison:

Number of LA Asbestos Structures Within Each Grid Opening - The inter-laboratory analysis performed by RESI identified four LA and one CH structures not identified by EMSL27 in the following GOs; A1-E8 (1 CH), A2-I8 (1 LA), A2-J7 (1 LA), A2-E11 (1 LA), A2-E13 (1 LA). Reconciliation was performed by RESI, which confirmed the presence of the missed structures. RESI also shipped the grids to EMSL Cinnaminson (EMSL04) in New Jersey, who also confirmed the presence of the structures not identified by EMSL27 during the re-preparation analysis. **Table 2** presents a GO by GO comparison between the two laboratories:

Table 2 - EPA Sample No. FB-00014 (Soil/Rock Flower Prep)							
	Interlab (RESI)			**Reprep (EMSL27)**			
GO	**LA**	**OA**	**CH**	**LA**	**OA**	**CH**	**Reconciliation Notes**
A1-D11	1	0	0	1	0	0	
A1-E8	1	0	1	1	0	0	Presence of CH structure confirmed by reconciliation performed by both RESI and EMSL04.
A1-F7	1	0	1	1	0	1	
A1-F15	0	0	0	0	0	0	
A2-F15	0	0	0	0	0	0	
A2-I8	2	0	0	1	0	0	Presence of LA structure confirmed by reconciliation performed by both RESI and EMSL04.
A2-J7	2	0	0	1	0	0	Presence of LA structure confirmed by reconciliation performed by both RESI and EMSL04.
A3-E11	2	0	0	1	0	0	Presence of LA structure confirmed by reconciliation

Table 2 - EPA Sample No. FB-00014 (Soil/Rock Flower Prep)							
GO	Interlab (RESI)			Reprep (EMSL27)			Reconciliation Notes
	LA	OA	CH	LA	OA	CH	
							performed by both RESI and EMSL04.
A3-E13	2	0	0	1	0	0	Presence of LA structure confirmed by reconciliation performed by both RESI and EMSL04.
A3-E15	1	0	0	1	0	0	

Asbestos Class of Structure - Both laboratories agreed on the assignment of amphiboles vs. CH, and amphiboles to LA or OA bins.

LA Structure Length - All fibers and bundles lengths agreed to within 1 micron (μm) or 10%, whichever was less stringent.

LA Structure Width - All fibers and bundles agreed to within 0.5 μm or 20%, whichever was less stringent.

Presence of Sodium (Na) and Potassium (K) – The EDXA spectra provided by both laboratories were in agreement regarding the presence or absence of Na and K, which for this sample was 100% XX (no Na or K detected) for both laboratories. However, a review of the spectra provided by both laboratories indicates that neither may have collected spectra for an appropriate amount of time. EDXA spectra from both laboratories were seldom collected for more than a few minutes and Silica (si) counts were consistently lower than the required 1,000 counts. RESI Si counts were as low as 40, with the majority below 500, and the EMSL27 Si counts were as low as 200 counts, with the majority below 400. These low Si counts may account for the observed absence of Na and K in the EDXA spectra for the identified LA structures.

Conclusion – The inter-laboratory analysis results of sample FB-00014 show that the asbestos class, length, and widths were consistent between the laboratories for common structures. However, and more significantly, the fact that EMSL27 missed five out of the 14 (36%) structures identified by RESI is cause for concern. This inter-laboratory analysis evaluation also revealed that neither EMSL27 nor RESI may be collecting EDXA spectra for a long enough period of time to accurately determine the presence of Na and K.

Hygeia/EMSL27 Inter-laboratory Analyses

On October 18, 2013 in response to direction from EPA, QATS selected several samples for a Hygeia/EMSL27 inter-laboratory study. Four samples were selected: FB-00004, FB-00009, AA-02777, and AA-02875. These samples were originally analyzed by Hygeia in 2013. Re-preparations were not performed for these samples; rather, ten GOs and the necessary alternate GOs were selected from the original “NOT QC” analysis of each of the four samples selected for Inter-laboratory analysis by EMSL27. The selection of GOs from the original “Not QC” analysis is a deviation from laboratory modification, but was done to expedite the process and reduce costs. On 10/31/2013, prior to shipping the TEM grids to EMSL27 to perform the inter-laboratory analyses, Hygeia provided the required paperwork (i.e. benchsheets and GO selection) to QATS for review and approval on 10/30/2013. Hygeia shipped the TEM grid preparations to EMSL27 on 10/31/2013. EMSL27 provided the inter-laboratory results on 11/14/2013. QATS compared the inter-laboratory results on a GO by GO basis and structure by structure basis to determine concordance using the same criteria applied above for inter-laboratory sample FB-00014. The following is a summary of the results for each of the four inter-laboratory sample analyses performed by EMSL27:

Number of LA Asbestos Structures within Each Grid Opening

FB-00004 – Eight LA structures identified by Hygeia in the original analysis were not identified by EMSL27 during the inter-laboratory analysis. LA structures by GO that were not identified by EMSL27 during the inter-laboratory analysis were: A1/G3-3 (1 LA), A1/G3-4 (1 LA), A1/E4-1 (1 LA), A1/G4-3 (3 LA), A2/E5-2 (1 LA), and A2/F4-3 (1 LA). During the reconciliation analysis performed by EMSL27, five NAM structures with similar length/width dimensions to that of the five LA structures identified by Hygeia were located. GO A1/G4-3 was damaged and could not be reconciled. **Table 3** presents a GO by GO comparison between the two laboratories:

Table 3 - EPA Sample No. FB-00004 (Soil/Rock Flower Prep.)

GO	Original Analysis (Hygeia)			Interlab (EMSL27)			Reconciliation Notes
	LA	OA	CH	LA	OA	CH	
A1/G3-3	2	0	0	1	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
A1/G3-4	3	0	0	2	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
A1/E4-1	3	0	0	2	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
A1/E4-2	3	0	0	3	0	0	
A1/G4-3	4	0	0	1	0	0	GO damaged, could not be reconciled
A2/E5-1	2	0	0	2	0	0	
A2/E5-2	2	0	0	1	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
A2/F4-3	3	0	0	2	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
A2/F4-4	3	0	0	3	0	0	
A2/E4-4	4	0	0	4	0	0	

FB-00009 – Five LA structures identified by Hygeia in the original analysis were not identified by EMSL27 during the inter-laboratory analysis. LA structures by GO that were not identified by EMSL27 during the inter-laboratory analysis were: B1/H3-2 (1 LA), B1/E4-3 (1 LA), B2/F3-2 (1 LA), B2/H3-1 (1 LA), and B2/C4-1 (1 LA). During the reconciliation analysis performed by EMSL27, one NAM structure with similar length/width dimensions to that of the LA structure identified by Hygeia was located. GOs B1/E4-3 and B2/C4-1 were damaged and could not be reconciled. **Table 4** presents a GO by GO comparison between the two laboratories:

Table 4 - EPA Sample No. FB-00009 (Soil/Rock Flower Prep)

GO	Original Analysis (Hygeia)			Interlab (EMSL27)			Reconciliation Notes
	LA	OA	CH	LA	OA	CH	
B1/F2-4	2	0	0	2	0	0	
B1/E2-3	2	0	0	2	0	0	
B1/H3-2	3	0	0	2	0	0	Reconciliation reported as ND
B1/E4-3	2	0	0	1	0	0	Could not be reconciled, GO damaged.

Table 4 - EPA Sample No. FB-00009 (Soil/Rock Flower Prep)							
	Original Analysis (Hygeia)			Interlab (EMSL27)			
GO	LA	OA	CH	LA	OA	CH	Reconciliation Notes
B1/G2-4	1	0	0	1	0	0	
B2/E3-2	2	0	0	2	0	0	
B2/F3-2	2	0	0	1	0	2	Reconciliation confirmed IL analysis.
B2/G3-1	1	0	0	1	0	0	Reconciliation confirmed IL analysis.
B2/H3-1	2	0	0	1	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
B2/C4-1	2	0	0	1	0	0	Could not be reconciled, GO damaged.

AA-02777 – One LA structure in GO D2/H4-1 that was identified by Hygeia in the original analysis was not identified by EMSL27 during the inter-laboratory analysis. During the reconciliation analysis performed by EMSL27, one NAM structure with similar length/width dimensions to that of the LA structure identified by Hygeia was located. **Table 5** presents a GO by GO comparison between the two laboratories:

Table 5 - EPA Sample No. AA-02777 (Air)							
	Original Analysis (Hygeia)			Interlab (EMSL27)			
GO	LA	OA	CH	LA	OA	CH	Reconciliation Notes
D1/F3-3	1	0	0	1	0	0	
D1/F4-3	1	0	0	1	0	0	
D1/G5-2	1	0	0	1	0	0	
D1/F4-2	0	0	0	0	0	0	
D1/G4-2	0	0	0	0	0	0	
D2/H4-1	1	0	0	0	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
D2/E4-1	0	0	0	0	0	0	
D2/G4-4	1	0	0	1	0	0	
D2/C4-3	1	0	0	1	0	0	
D2/F5-1	0	0	0	0	0	0	

AA-02875 – Three LA structures identified by Hygeia in the original analysis were not identified by EMSL27 during the inter-laboratory analysis. LA structures by GO that were not identified by EMSL27 during the inter-laboratory analysis were: C1/E6-2 (1 LA), C1/E4-1(1 LA), and C2/K4-4 (1 LA). During the reconciliation analysis performed by EMSL27, one NAM structure with similar length/width dimensions to that of the LA structure identified by Hygeia was located. Note that three LA structures identified by EMSL27 during the inter-laboratory were not identified by Hygeia in the original analysis. **Table 6** presents a GO by GO comparison between the two laboratories:

Table 6 - EPA Sample No. AA-02875 (Air)							
	Original Analysis (Hygeia)			Interlab (EMSL27)			
GO	LA	OA	CH	LA	OA	CH	Reconciliation Notes
C1/E6-2	1	0	0	0	0	0	A NAM structure with close length/width dimensions located during the reconciliation.
C1/G5-1	0	0	0	0	0	0	
C1/G4-4	0	0	0	0	0	0	
C1/E4-1	1	0	0	0	0	0	IL results confirmed by reconciliation.
C1/E4-2	1	0	0	1	0	0	
C2/F4-1	1	0	0	2	0	0	IL results confirmed by reconciliation.
C2/K4-4	1	0	0	0	0	0	IL results confirmed by reconciliation.
C2/H4-4	1	0	0	2	0	0	IL results confirmed by reconciliation.
C2/K5-1	0	0	1	1	0	0	IL results confirmed by reconciliation.
C2/F5-3	1	0	0	1	0	0	

Asbestos Class of Structure - Both laboratories agreed on the assignment of amphiboles versus CH, and amphiboles to LA or OA bins for all four samples.

LA Structure Length – Slight structure length reporting anomalies in single structures in three of the four inter-laboratory samples were observed:

- FB-00004 – One LA structure did not meet the length criteria; however, the structure terminated in a non-countable grid bar for the inter-lab analysis and the length was doubled, which accounts for the difference. A review of the structure sketches on the bench sheets indicates the structure has moved into the grid bar over time, and therefore both measurements are accurate.
- FB-00009 – One structure, a LA structure identified by both laboratories in GO B1/E2-3, did not meet the length criteria of ≤ 1µm or ≤ 10% difference.
- AA-02777 - One structure, a LA structure identified by both laboratories in GO D1/G5-2, did not meet the length criteria of ≤ 1µm or ≤ 10% difference.
- AA-02875 - All concordant.

LA Structure Width - All fibers and bundles agreed to within 0.5 µm or 20%, whichever was less stringent, for all of matched structures in all four of the samples.

Presence of Sodium (Na) and Potassium (K) – EMSL27 reported far fewer spectra containing Na and K than Hygeia in the four inter-laboratory samples analyzed. **Table 7** provides a summary of the EDXA results for the LA structures identified for each sample:

Table 7 - Laboratory EDXA Spectra Comparison					
		EDXA Observation			
Sample No	Laboratory	NaK	NaX	XK	XX
FB-00004	Hygeia	29	0	0	0
	EMSL27	5	1	0	16
FB-00009	Hygeia	17	1	0	1
	EMSL27	1	0	0	13
AA-02777	Hygeia	6	0	0	0

Table 7 - Laboratory EDXA Spectra Comparison					
Sample No	Laboratory	EDXA Observation			
		NaK	NaX	XK	XX
	EMSL27	0	1	0	4
AA-02875	Hygeia	5	0	0	3
	EMSL27	0	0	1	5
Totals	**Hygeia**	**57**	**1**	**0**	**4**
	EMSL27	**6**	**2**	**1**	**38**

Hygeia reported 57 spectra as NaK versus 6 for EMSL27, and EMSL27 reported 38 spectra as XX (no Na or K) versus 4 for Hygeia.

Note that prior to the training provided to the EMSL27 staff by ESAT Region 8 on September 25-26, 2013, EMSL27 consistently reported NaK (WRTA), but seldom reported XX. However, this most current study indicates a potential reversal of that trend with XX being reported at a high frequency.

A review of the spectra provided by EMSL27 found that of the 30 spectra provided for structures determined to be LA structures, only seven (23%) had Si peaks that met or exceeded the 1,000 count criteria. A review of the spectra provided by Hygeia found that of the 34 spectra provided for LA identified structures, 17 (50%) had Si peaks that met or exceeded the 1,000 count criteria. Note that the detector on the Hygeia TEM is equipped with a high angle detector with a Beryllium window which may be more sensitive than the detector utilized by EMSL27. EMSL27 uses a detector that also has a beryllium window, but it is a horizontal detector.

Further investigation into those structures for which spectra that were available from both of the laboratories indicates that short spectral collection times can influence the amount of Na and K detected. Note that in **Table 8** for those samples with matching or partially matching spectra (highlighted), the Si peaks have roughly the same number of counts. However, the spectra for XX values reported by EMSL27 that were reported as NaK by Hygeia almost always have lower Si counts.

The results in the table below suggest that had EMSL27 collected spectra for a longer period of time, Na and K peaks could possibly have been identified; however, this observation is preliminary. More information needs to be gathered concerning detectors, etc., before a conclusion regarding spectral reporting can be made.

Table 8 - EDXA Silica Counts													
	Grid/GO		EMSL27					Hygeia					
Sample No.	Grid	GO	Str	Class	Desc	EDXA	Si Peak	Str	Class	desc	EDXA	Si Peak	NaK match
AA-02777	D1	F3-3	1	LA	AT	XX	400	1	LA	WRTA	NaK	555	No
AA-02777	D1	F4-3	2	LA	WRTA	NaX	400	2	LA	WRTA	NaK	476	Partial
AA-02777	D1	G5-2	3	LA	AT	XX	450	3	LA	WRTA	NaK	965	No
AA-02777	D2	G4-4	4	LA	AT	XX	100	6	LA	WRTA	NaK	654	No
AA-02875	C1	E6-2	0	NAM	UN	XX	200	1	LA	WRTA	NaK	1130	No
AA-02875	C1	E4-2	1	LA	AT	XX	1700	6	LA	TR	XX	1064	Yes
FB-00004	A1	G3-3	1	LA	WRTA	NaX	1100	2	LA	WRTA	NaK	927	Partial
FB-00004	A1	G3-4	2	LA	AT	XX	300	4	LA	WRTA	NaK	1028	No

Table 8 - EDXA Silica Counts													
	Grid/GO		EMSL27					Hygeia					
Sample No.	Grid	GO	Str	Class	Desc	EDXA	Si Peak	Str	Class	desc	EDXA	Si Peak	NaK match
FB-00009	B1	F2-4	1	LA	AT	XX	300	4	LA	WRTA	NaK	645	No
FB-00009	B1	F2-4	2	LA	WRTA	NaK	1000	3	LA	WRTA	NaK	864	Yes
FB-00009	B1	E2-3	3	LA	AT	XX	800	5	LA	WRTA	NaK	697	No
FB-00009	B1	E2-3	4	LA	AT	XX	275	6	LA	WRTA	NaK	779	No

Conclusion – A total of 17 LA structures detected by Hygeia during the original analysis of the four samples above were not detected by EMSL27 in either the inter-laboratory or reconciliation analyses. This concludes that either A) the structures were not present during the original analysis (highly doubtful); B) the grid preparations were damaged in such a way that structures were lost; or C) the structures are present, but were identified in neither the inter-laboratory or reconciliation analyses. Concerning the detection of Na and K from EDXA analysis, there appears to be some evidence that EMSL27 is not collecting spectra for an adequate amount of time.

Final Conclusions and Recommendations

The results from both of the above IL studies, one between EMSL27 and RESI, and the other between EMSL27 and Hygeia, indicate that EMSL27 has yet to demonstrate the necessary proficiency to accurately analyze Libby samples for LA. The number of potentially missed LA structures from the two IL studies, 22 total, is significant. In addition, EMSL27 consistently fails to follow the directions provided in laboratory modification LB-000029. Additional training for this laboratory is recommended. Another recommendation based on the results of these ILs is that all laboratories be further trained regarding the collection of EDXA spectra to ensure the detection of Na and K when present. Prior to that it might be useful to compile a list of the types of EDXA detectors used by each of the laboratories to evaluate whether detector type (i.e., high angle versus horizontal) has any bearing on the detection of Na and K.